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# THE MOISTURE PROTECTION OF STRONG OPTICAL FIBERS

Gulf & Western Applied Science Laboratories



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# THE MOISTURE PROTECTION OF STRONG OPTICAL FIBERS

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This program addresses the military and commercial needs for strong optical fibers having a high degree of durability. The approach to solving this problem is to demonstrate the feasibility of producing hermetically coated fibers, coating with a suitable hermetic barrier, proof testing, optical and mechanical characterization, and analysis of results.

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and built by Applied Science Laboratories (ASL), the prime contractor. It has been used to coat fibers which have previously been drawn by the subcontractor, Galileo Electro-Optics Corporation (GEOC). Materials evaluated as hermetic coatings included ion deposited carbon, tin, and indium. Initial results indicate that the ion deposited forms of carbon and indium alone or in combination appear to provide the best compromise of coating properties, while not degrading fiber strength. While this work was in progress by ASL, Galileo was constructing a dedicated $CO_2$ laser draw tower which could provide a suitable in-line interface to the hermetic coating system. The ASL hermetic coating system was then interfaced to the GEOC draw tower at which time fibers were drawn and coated in 100 meter lengths. The results of these tests will be available upon completion of various static and dynamic fatigue evaluations as well as the optical characterization.

The long range objective of this program is to produce fibers with 1-7 db per kilometer attenuation at 850 nanometers, proof tested in excess of 100,000 psi, in lengths of 10-30 kilometers. It is anticipated that after hermetic coating, these optical waveguides will exhibit a 95% probability of survival in moist environments when subjected to a 2% continuous strain over periods up to 10 years.

Previous work reported by others has indicated the difficulty of preventing fiber failure in humid environments. The strength of plastic jacketed optical waveguides quickly degrades over a period of days due to moisture migration through the loosely packed polymer matrix (Kao, 1980) (Midwinter, 1979). As a result, the fibers fail due to the interaction of $H_2O$ with microscopic flaws on the fiber surface. Through careful control of processing parameters and starting materials, very strong ( 2,000 MPa) fibers may be drawn. It has been shown that such strengths may be preserved through in-line dip coating of the fiber with a metal, e.g. aluminum (Pinnow, 1977). However, such metal clad fibers exhigib dynamic fatigue and stress discontinuities in the jacket which results in premature fiber failure, and microbending losses.

This program is therefore directed to developing a hermetic coating technique which greatly reduces the development and growth of surface stress discontinuities and crack propagation. The following sections detail the techniques and results involved in implementing ion deposited coatings for hermetic protection of laser drawn optical fiber.

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## ACKNOWLEDGEMENT

This program is sponsored by Solid State Sciences Division, Rome Air Development Center (AFSC), and by Defense Advanced Research Projects Agency, under Contract No. F19628-78-C-0180.

The prime contractor is Gulf + Western Applied Science Laboratories, and the subcontractor is Galileo Electro-Optics Corporation.

The principal investigator at Applied Science Laboratories is Dr. S. Aisenberg and the program manager is Mr. M. Stein.

The principal investigator at Galileo is Dr. R. Jaeger, and the program manager is Mr. J. Stevens.

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## I. SUMMARY

This program addresses the military and commercial needs for strong optical fibers having a high degree of durability. The approach to solving this problem is to demonstrate the feasibility of producing hermetically coated fibers. This includes the drawing of high strength, low loss fibers, coating with a suitable hermetic barrier, proof testing, optical and mechanical characterization, and analysis of results.

At present in the program, the hermetic coating system has been designed and built by Applied Science Laboratories (ASL), the prime contractor. It has been used to coat fibers which had previously been drawn by the subcontractor, Galileo Electro-Optics Corporation (GEOC). Materials evaluated as hermetic coatings included ion deposited carbon, tin, and indium. Initial results indicate that the ion deposited forms of carbon and indium alone or in combination appear to provide the best compromise of coating properties, while not degrading fiber strength. While this work was in progress by ASL, Galileo was constructing a dedicated $CO_2$ laser draw tower which could provide a suitable in-line interface to the hermetic coating system. The ASL hermetic coating system was then interfaced to the GEOC draw tower at which time fibers were drawn and coated in 100 meter lengths. The results of these tests will be available upon completion of various static and dynamic fatigue evaluations as well as the optical characterization.

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careful control of processing parameters and starting materials, very strong (>2,000 MPa) fibers may be drawn. It has been shown that such strengths may be preserved through in-line dip coating of the fiber with a metal, e.g. aluminum (Pinnow, 1977). However, such metal clad fibers exhibit dynamic fatigue and stress discontinuities in the jacket which results in premature fiber failure, and microbending losses.

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## II. INTRODUCTION AND BACKGROUND

### A. Program Scope

It has been found that strong fibers with proof strengths of several hundred thousand psi may be drawn by carefully controlling the starting materials and processing parameters. However, the probability of failure is not only stress and strain dependent, but is also time dependent. The physical mechanism responsible is crack growth under the combined effects of humidity and strain. Cracks propagate with time due to chemical erosion under stress at the crack tip (Kao, 1980). Thus, we can expect increased lifetimes under stress if moisture is kept from the fiber surface. Previous work has indicated the inadequacy of polymers as migration barriers, and some metal coated fibers exhibit accelerated cyclic fatigue failure as well as inducing excess attenuation (Pinnow, 1977).

The hermetic coating process developed under this program is intended to achieve high strengths and long lifetimes in fibers by depositing, in-line, thin layers of diamond-like carbon and indium on the freshly drawn fibers.

The construction of a dedicated $CO_2$ laser draw tower and hermetic coating system constitute a major phase of this program. To achieve the program objective at reduced cost to the government, over half of the capital equipment requirements are being met by the contractor, ASL, and the subcontractor, GEOC.

Upon completion of the coating system at ASL, it was moved to GEOC and installed on the laser draw tower. When system start-up and performance verification are complete, a series of coating runs will be made to optimize coating performance.

### B. Fiber Optic Technology

Since the early 1970s when it became apparent that the attenuation criterion of 20 db per kilometer or less could be met, many fiber optic fabrication processes have been employed for communication purposes. This achievement motivated workers at Bell Telephone Labs to explore

other processes for development of high strength, low loss fibers. One of the more promising techniques developed by Ray Jaeger (Jaeger, 1976) and others was the generation of laser drawn fiber. It was found that the highest numerical apertures and lowest losses were achieved when the fiber was fabricated using a $CO_2$ laser drawing process. It was also recognized that, although characteristics of the polymer coated laser drawn fiber would meet the needs of a communications system, stress corrosion effects due to water and ion migration can lead to strength reduction.

When Dr. Jaeger joined GEOC, he transferred the laser drawing technology to that facility, and has duplicated and simplified the process. The current effort emphasizes protection of the pure silica surface of silica fibers drawn by the $CO_2$ laser drawing process. This permits, for reasons of economy, development work based on solid silica fibers that do not have waveguide properties. However, fiber waveguides will subsequently be fabricated and coated to verify that their strength and optical loss is not significantly influenced by the ion-plasma coating procedure, and that the hermetic coating preserves the strength.

## C. Ion Deposition Technology

In the early 1970s, while the fiber optic work was in progress at Bell Labs, S. Aisenberg and R. Chabot (Aisenberg, 1971) had developed an ion beam deposition technique to generate thin films of diamond-like carbon. It was established that the carbon films deposited by this technique are insulating and have characteristics similar to that of carbon in the diamond form. Key properties were transparency, refractive index greater than 2, scratch resistance, resistance to attack by strong acids and bases, as well as resistance to moisture migration and ion diffusion. A more detailed summary of the observed properties of diamond-like carbon films is given in Appendix A.

With the development of more stringent military demands for fiber optic communication systems, this unique coating appeared to be a candidate for protection of freshly drawn pristine fibers. In addition to the diamond-like form of carbon, other materials as well may be

deposited by the ion-plasma beam technique. It was found that by simple modification of electrode geometry, the technique could be used to put the coatings on in-line under vacuum. This contract, initiated by ARPA funding, involves the construction of the apparatus, evaluation of the coating technique, and evaluation of the coatings produced, and of the strong optical fibers that are coated.

## III. TECHNICAL ACCOMPLISHMENTS

### A. Program Objectives

Gulf + Western Applied Science Laboratories (ASL) recognizes that successful application of multi-kilometer lengths of optical communication fiber for a variety of military applications requires that the fiber withstand high level short duration stress and exhibit a very high survival probability for long periods of time in a variety of adverse environments. Estimated values of short term stress levels may approach 500,000 psi (5% strain for several seconds) while long term stress levels may be in excess of 300,000 psi (3% strain). Also, high level variable frequency cyclical stress may be encountered in certain military environments. Equally important is the requirement that the transmission characteristics be essentially unaffected by mechanical stress, temperature, pressure, irradiation, and exposure to corrosive chemicals.

During the past several years, significant advances in the short term mechanical strength of optical fiber cable has been achieved. Plastic coated fibers are presently being manufactured that have passed proof test stress levels in excess of one hundred thousand psi in kilometer lengths. This advance is attributed to improvements in preform substrate quality, fabrication procedures and subsequent preform surface treatments, drawing techniques and finally, coating techniques. However, no matter what the short term mechanical strength is, these fibers still exhibit the same stress corrosion characteristics (static fatigue phenomena) as uncoated fiber.

Fiber coating hermeticity is the key to eliminating this time dependent, stress induced environmentally accelerated strength deterioration. The attainment of truly hermetic coatings for strong optical fibers is vital in the meeting of optical fiber specifications required for present and future military and commercial applications and is the primary goal of this program. Based on the prliminary work done with the apparatus used on this program, the contractors anticipate demonstrating the value of energetic ion deposition as a hermetic coating technique capable of meeting the above requirements.

The prime objective of this program is the development of strong optical fibers having a high degree of durability. Previous work has shown that optical waveguides possessing suitable attenuation and proof strength can be fabricated by several techniques. However, static fatigue associated with the presence of water causes the initial strength to decline with time. This can be prevented if the fiber is hermetically sealed by coating immediately after the drawing process. Besides providing a continuous pinhole-free hermetic barrier, the coating should maintain its integrity for 10 years use life in the presence of moisture. It should also not be degraded by abrasion or cyclic fatigue. Additionally, the fiber should survive the coating process without strength degradation.

Some potential problems and solutions associated with the plasma-ion deposition process for hermetic coatings are discussed in Appendix B.

## B. Materials

The initial portion of this program emphasized the selection and validation of candidate coating materials. In selecting a suitable hermetic material, initial consideration is given to vapor permeability, which can be defined as the product of solubility of vapor in the material and its diffusion coefficient. There are several materials which exhibit very low solubility for water vapor. Materials with the best values are some metals and several dielectrics including glass. Historically, polymer coatings have been used for fiber protection. Due to the general open nature of the polymer matrix, however, solubility and diffusion are much higher than for metals and dielectrics. Consequently, the primary hermetic materials were selected from each category; low permeability dielectrics and metals.

Instead of trying to select the best material, it was decided to try a combination from each category. The coating structure would then include a base coating of a dielectric, a metal clad, and a polymeric overcoat for abrasion resistance.

The ion deposited carbon film has been selected as a dielectric based on work at ASL which has been developing the ion-plasma deposition technique for the past 10 years. The technique involves the use of ion

milling during deposition to selectively permit stronger carbon-carbon bonds to form. As a result, the film tends to be polycrystalline in form with a very tight lattice. Results based on X-ray analysis have shown the material to possess a microcrystalline diamond-like structure (Aisenberg, 1971) (Spencer, 1976). A summary of the general properties and characteristics of thin film diamond-like carbon is contained in Appendix A.

In the current program, the energetic ion-plasma deposition technique was employed to coat fibers in-line directly after drawing. Primary advantages gained by the material and process are a tight hermetic coating with few problems resulting from contamination due to the high vacuum levels maintained during deposition. The material can also be put down with excellent hermetic characteristics and thicknesses of 0.1 microns. This thickness is desirable to eliminate microbending stresses and differential thermal stressing with respect to the glass substrate.

The key property to selection of the metal is a tight lattice with low vapor permeability and high ductility which would not induce microbend stressing, as well as the absence of other anomalies which could induce stress discontinuities.

In considerating various metallic materials, the work of others has been reviewed. Much work has been done by Pinnow of Hughes Research Labs. in this area (Pinnow, 1977). His findings indicated that aluminum tended to induce micro-stressing resulting in early cyclic fatigue failure from slip plane dislocations. This takes the form of extra optical losses due to microbending. Other materials also possess problems. Tin, for example, has been shown to possess 2 allotropic forms with a recrystallization temperature within the military use range. Indium was selected as the initial material for evaluation in this work since it had no allotropic forms in the intended temperature range, and was sufficiently ductile to permit repeated stress relaxation cycles without memory. A summary of the related properties of indium is provided in Appendix C.

Initial experimental work began with ion deposition of tin and indium for comparison of adhesion and properties. Results from glass slide tests indicated that the adhesion was adequate for both materials

when the energetic ion deposition technique was employed. Indium, with a compressive strength of only 310 psi, appeared to bond very well to glass. Its adhesive strength, in fact, appeared to be greater than the cohesive strength of the indium itself in many cases. Due to its softness, however, care must be taken to protect the thin coating once it is put down. The intended coating thickness range will probably be on the order of a micron or less.

While indium can provide a tight hermetic coating without creating stress discontinuities, it is also easily abraded due to its low ductility and strength. To avoid this problem, a UV curable polymeric overcoat will be applied after the hermetic coating stage, and before the pinch-wheels. The polymer coating is specifically designed for fibers and has been used successfully by GEOC on bare silica fibers. This overcoat is intended to provide physical protection of the hermetic coatings as well as to prevent ionic interaction with the metallic layer when the fiber is immersed in water.

Diamond-like carbon and indium have been selected for this program. Other materials can also be applied by the ion-plasma process.

## C. Hermetic Coating Apparatus

The hermetic coating process occurs in vacuum (immediately after drawing) within a module equipped with differentially pumped entrance and exit ports. Once inside the coating chamber, the fiber is exposed to plasma-ion bombardment to clean the fiber surface of adsorbed gases and debris. The fiber then passes through two energetic ion deposition stages. Each stage can be used for diamond-like carbon deposition or for deposition of other dielectrics. The second stage is also equipped for metal deposition. Major emphasis was placed on indium for reasons explained in the materials section of this Report. Other metals, however, can be substituted.

The coating system consists of a 6-way 6-inch diameter glass cross fitted with fiber entrance and exit ports, a 6-inch oil diffusion pump, and feed throughs for power, control, and cooling. The system is powered and controlled by a console which maintains compiete control of

the system as well as of the mechanical pumps used for the differentially pumped entrance and exit ports.

The current coating apparatus, as shown in Figure 1 consists of a series of 6-inch glass crosses and tees. These units are mounted with adequate flanges for feed throughs and control, on a 2-inch thick aluminum plate. This plate serves as a base support and framework for the pumping assembly as well as for the coating chamber. The coating system is fitted with controls for measuring various pressures as well as a number of valves for pump control and gas feed. The control unit is mounted in the same framework as the plasma power supplies.

The system schematic can be seen in Figure 1. A pictorial view showing the various components is contained in Figure 2; Figure 3 is a photograph of the current system.

The fiber must pass from the ambient environment into a vacuum chamber maintained at several microns pressure without introducing any air contamination. Likewise, the fiber must again exit to atmosphere after passing through the various coating stages. The differentially pumped entrance and exit ports consist of a series of pumping baffles. Each baffle plate has an orifice of controlled dimensions.

Mechanical vacuum pumps reduce the pressure at each differentially pumped stage. The first baffle reduces the pressure from atmosphere using a large pump. Subsequent baffles use two smaller pumps to achieve the desired pressure prior to entering the coating chamber which is operated at about 30 microns pressure. The lower pumping manifold is coupled to the same large pump as is the top manifold. Additionally, a small pump is used to produce the pressure drop at the inner baffle at the bottom port.

Another requirement of the coating system is to keep air contamination out of the ion-plasma deposition regions. Therefore, the top and bottom pumping stacks are equipped with argon shrouds or buffer chambers. A liquid argon dewer has been selected as a gas source since the gas stream will be maintained relatively clean and water-free. Many materials, including water, are of course, frozen at these two temperatures. Additionally, the slow, large surface evaporation rate of the liquid argon prevents entrainment of contaminants in the gas

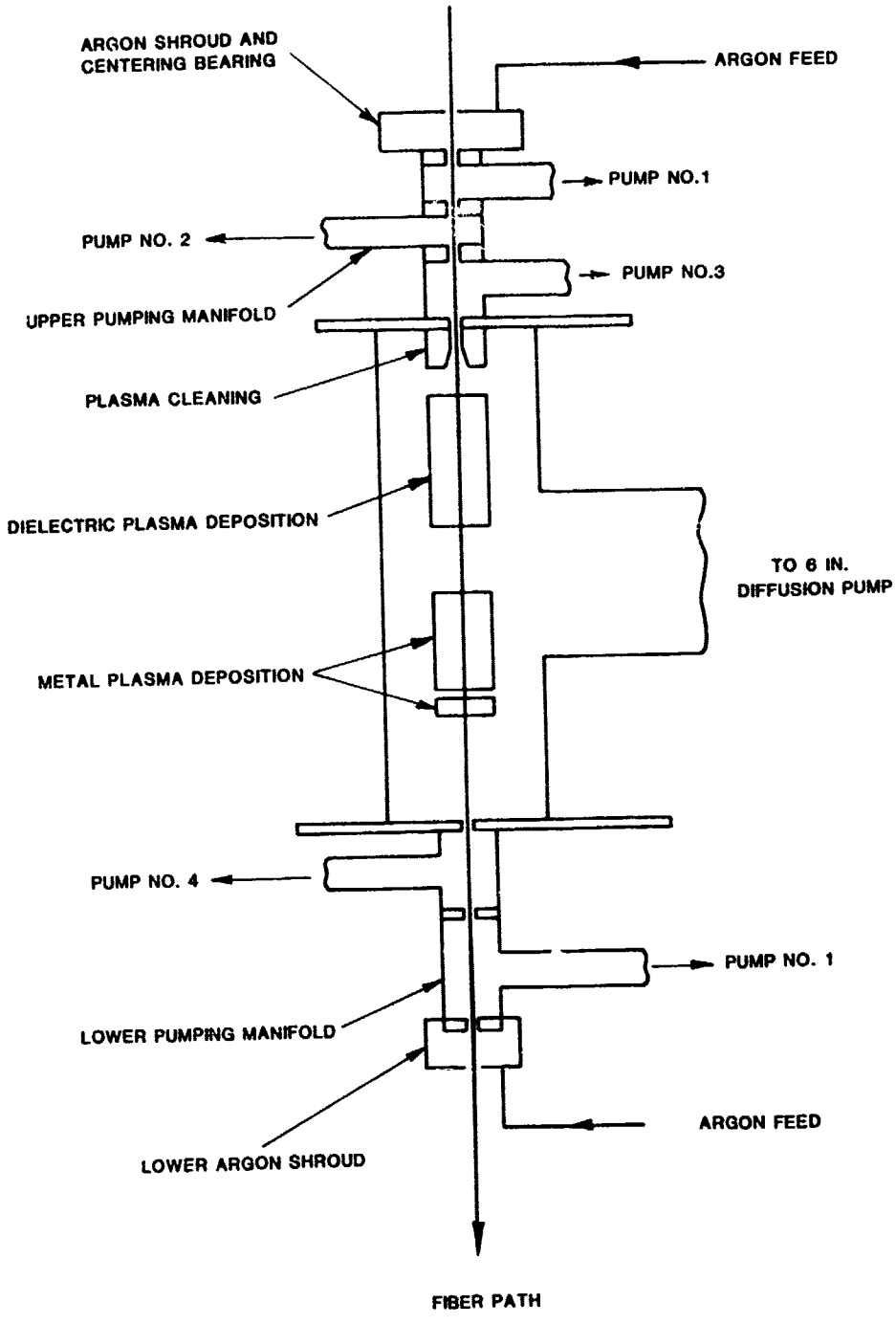


Fig. 1 Hermetic Coating System

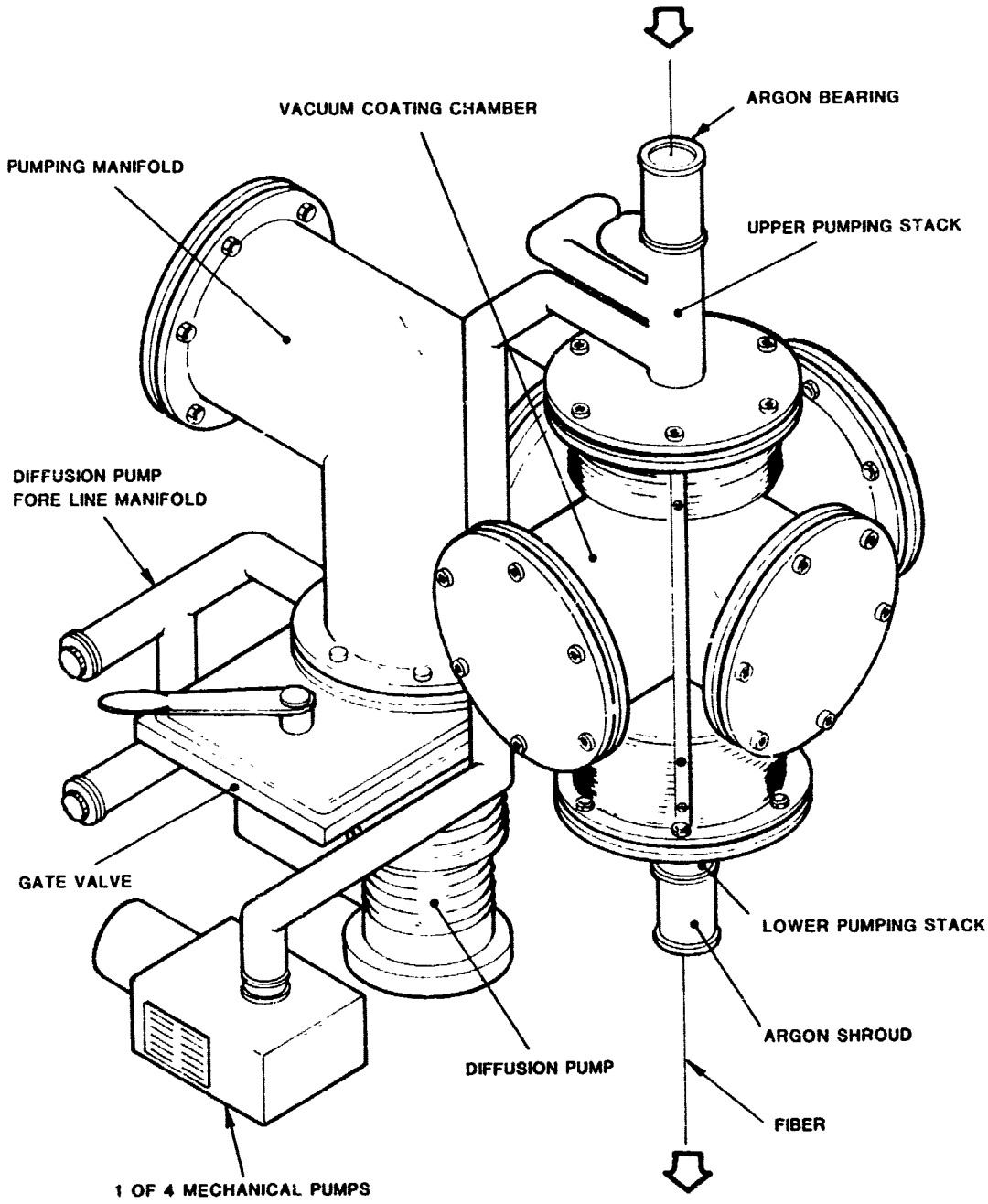


Fig. 2 Physical Construction of Hermetic Coating System

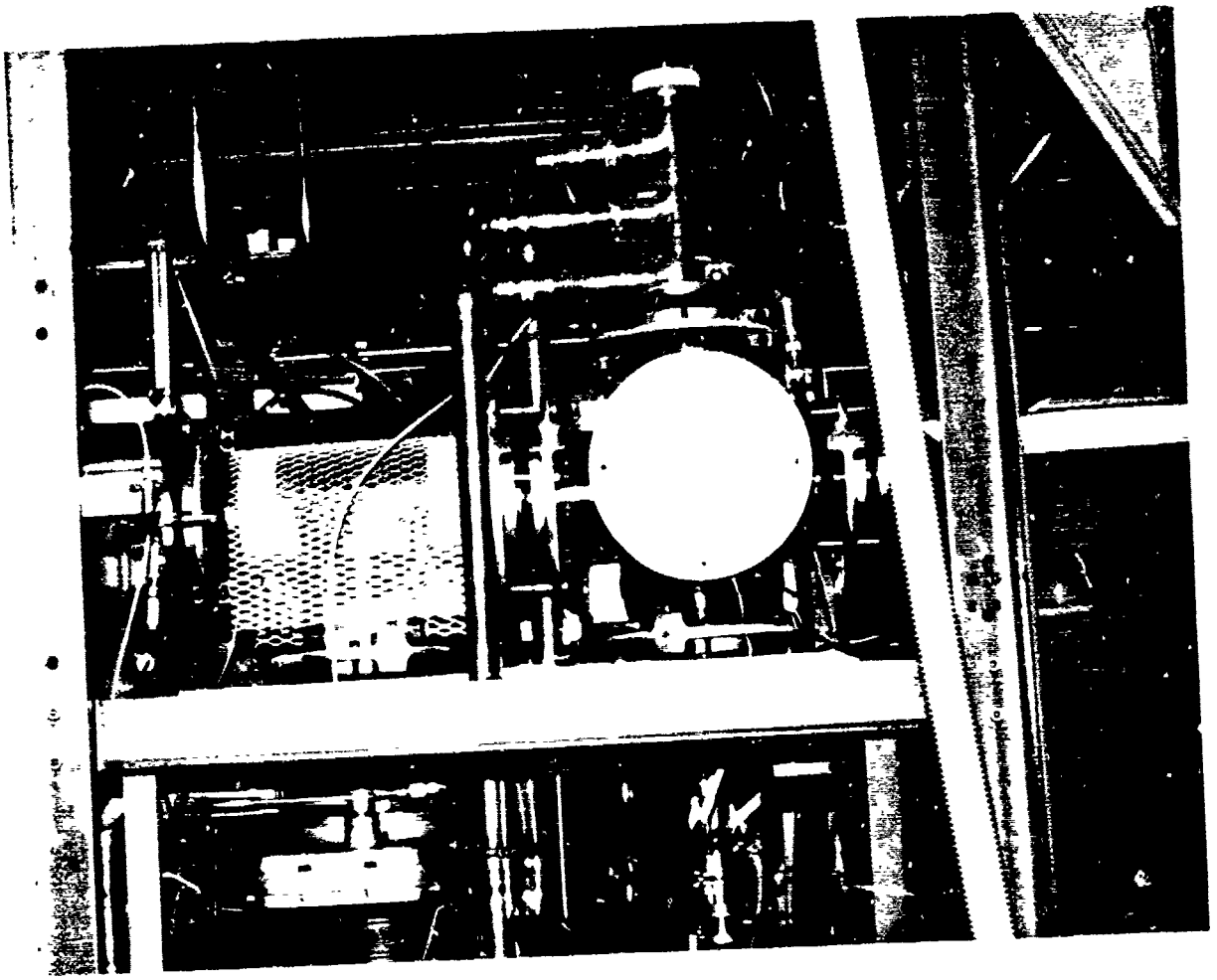

Fig. 3 View of Hermetic Coating System

stream. Each argon shroud is adjusted to provide a slightly positive out-flow of gas along the fiber path. Argon gas, therefore, flows both out of the argon buffer chamber and into the vacuum system.

An additional feature of the ports is the use of gas flow to help keep the fiber centered in the port openings. Various flow geometries are being investigated.

The vacuum level in the actual coating chamber is maintained using a 6-inch oil diffusion pump. The pump, in conjunction with a water-cooled baffle, is used to provide the desired deposition conditions while maintaining a clean atmosphere. Due to the argon influx rates used to admit the fiber to the system, the diffusion pump operates at a very high throughput. At these rates, the manufacturer, Varian, has been unable to measure oil backstreaming rates. Therefore, we feel the coating atmosphere to be sufficiently clean to produce a good quality coating.

1. Plasma Cleaning Stage

Upon entering the actual deposition chamber, the first stage encountered by the fiber is a plasma cleaning stage. The objective of this stage is to bombard the fiber with an ion flux to produce a degree of surface cleaning and scrubbing of contaminants as well as of adsorbed gases. Prior experience with deposition techniques has shown that it is not uncommon to find 50-100 Angstroms of adsorbed gases and contaminants on a "clean" surface in vacua. Past tests have shown that the plasma cleaning technique on glass is indeed effective. It is simple to observe that a glass slide wets completely upon being exposed to a plasma cleaning process, whereas prior to plasma cleaning, spotty, inadequate wetting is typically noted. The plasma cleaning process itself consists of an argon plasma jet formed by the gas feed entering the high vacuum coating system. This jet is maintained by the electrode geometry which defines the plasma zones. Additionally, a magnetic field is applied axially to the fiber which confines the excitation zone about the fiber and cleaning electrodes. The ion density in

this zone is sufficient to produce scrubbing prior to entering the dielectric deposition stage.

2. Deposition Stages

The dielectric plasma deposition electrode is directly below the cleaning stage. The deposition electrode currently being used is fabricated from graphite. Graphite has been selected as a source of carbon ions for the present deposition technique. When other dielectric materials are investigated, this electrode will be fabricated from the intended deposition material. Consequently, changing deposition materials becomes rather straightforward.

The dielectric plasma electrode is designed as a hollow cathode cylindrical magnetron. The fiber enters an aperture through one end of the cylinder through an anode ring. As it passes through the hollow cathode a negative potential is developed on the fiber surface due to its insulating characteristics. This potential is on the order of minus several hundred volts. In the confined cathode zone, electrode bombardment is enhanced by trapped electrons which maintain the primary plasma. Additionally, the applied axial magnetic field transforms the electron trajectory into a spiral path increasing the probability of ionization, hence increasing the sputter yield at lower pressures. As a result of these efforts, the sputter yield has been enhanced greatly over alternate beam techniques employed by ASL. In addition to a high sputter yield, the major factor for using this type of electrode geometry is to achieve growth and coalescence of a diamond-like carbon film. Previous work indicates that film growth can generally occur by growth coalescence and mobility coalescence (Lewis, 1978) (Vossen, 1978) (Holland, 1966). In a straight evaporation process, growth coalescence usually dominates below 500°C This is when initial deposition islands tend to increase in size until the islands actually touch causing coalescence on the surface. Mobility enhanced coalescence, however, can greatly enhance the tightness and pinhole-free characteristics of the

film while causing coalescence to occur much earlier in the deposition process. For these reasons, we have employed the plasma technique which relies on electrode and ion bombardment of the fiber substrate. In this way, the fiber is cleaned of weaker bonds and adsorbed gases while a tight lattice, in the case of carbon, is permitted to build. Prior techniques with other dielectrics, i.e., boron nitride, have indicated a similar smooth film forms readily. The high surface energy can be achieved on the fiber while maintaining the fiber temperature at reasonable temperatures, i.e., 300°C. At draw rates of 2 meters/minute, a film thickness of approximately 300 Angstroms of carbon builds up in the first dielectric deposition electrode.

The next ion-plasma stage can be used for metal deposition. Again, a very similar technique is employed as was used for the carbon electrode. The metal presently being deposited is indium. This appears desirable due to its good hermeticity and wetting characteristics. For this electrode, a support material is coated with a thick indium layer. As the fiber passes through this stage, a similar power supply operates the plasma to produce the high sputter yield from the walls. In order to achieve the high deposition rate required to build a 2 micron thick coating onto the fiber, evaporation is also employed.

Evaporation is a good intense source of thermal metallic atoms. These atoms are produced from a cylindrical heat source. The fiber passes through a central hole in the source from which metal is being evaporated at a rapid rate. The metal atoms travel by line of sight into the metal plasma deposition chamber directly above it. In this chamber, the metal atoms are strongly ionized and behave much the same as the ions do in the first dielectric coating chamber. Hence, the initial layers of metal are deposited by ion deposition onto the fiber. As the fiber passes through the metal evaporation filament opening, additional monolayers are built up rapidly by direct evaporation. The filament also serves as a source of heat to enhance coalescence and flow. The present apparatus uses a fixed metal supply in the evaporant source, and

can only coat moderate lengths of fiber (100 meters) before the charge is exhausted.

The system currently being used was designed for experimentation with the greatest versatility to permit convenient changing of materials and deposition parameters. Based on in-line results being obtained, parameters and materials will be identified which should yield optimal coating performance. A production version of this initial design is intended to interface with standard existing draw towers.

D. Laser Draw Tower

The drawing of optical fiber whose strength is invariant with time when exposed to adverse environments requires a hermetic coating that is concentric about the fiber, continuous over the entire length of the fiber (no pinholes), and is of constant and uniform thickness. To produce coatings to these specifications, fiber must be fed through the Hermetic Coating System (HCS) at a constant and compatible rate (1 meter/min to 10 meters/min) and the diameter of this fiber precisely controlled (to $\pm$1%). In addition, the hermetically coated fiber should be externally buffered to protect the integrity of the thin hermetic coating and to aid in its spooling and further handling.

Galileo has built a laser draw tower, illustrated in Fig. 4, that will produce fiber to the above requirements. This tower is a composite of several towers that have been built by Galileo. It was designed to minimize the effects of external vibration and to allow easy access to the HCS from all sides. The precision draw and feed mechanisms are coupled in a feedback loop to a diameter monitoring device. Similar control systems, presently in operation, have produced fiber to better than $\pm$1% diameter tolerances at the specified feed rates. The $CO_2$ laser beam delivery system has been built to produce a stable heat zone which can be intentionally varied to produce different heat zone geometries which will facilitate the drawing of IVPO (inside vapor phase oxidation) fiber. (French, 1979). The lower tower structure is prismatic in shape. Its base is an equilateral triangle having 8' long sides and it is 8' 7" high.

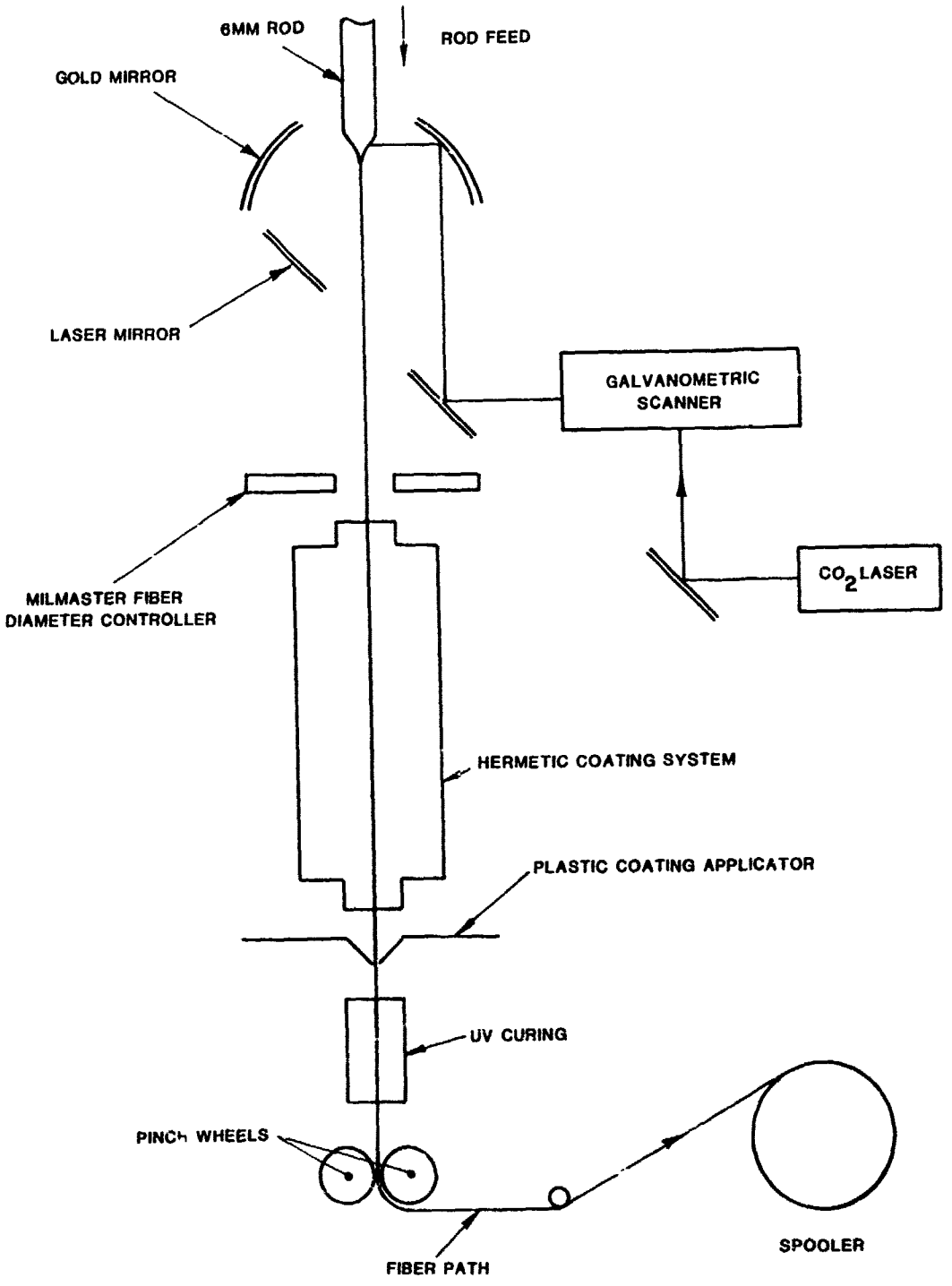


Fig. 4 In-Line Arrangement of Laser Draw Tower and Hermetic Coating System

The three upright corners are constructed using 4" diameter steel conduit that are vibrationally isolated from the floor. This structural shape was chosen for its inherent stability since it will house the HSC. The fiber draw axis is approximately one foot within the triangular structure to reduce moment effects. The UV curing system and draw mechanism are mounted on a cross brace arrangement below the HCS cavity.

The upper tower structure is made of sheet metal and angle iron. It supports the feed mechanism, the conical mirror, part of the beam delivery system, and the diameter monitoring device.

Both the preform feed mechanism and the fiber draw mechanism use a special design control system. The heart of this system is a DC direct drive servomotor. The motor for the feed system is directly coupled to a precision feed screw and operates between 1 and 100 revolutions per hour in the down feed position and at 200 revolutions per minute in the reverse position.

The motor for the draw system is directly coupled to a 3" diameter pinch wheel and is designed to operate between 0 and 400 RPM.

Both systems can be operated simultaneously or independently of each other in either a manual mode or in a feedback loop. The feedback loop utilizes a DC voltage signal provided by the diameter monitoring device which is added to or subtracted from the motor's voltage as the diameter increases or decreases.

A MilMaster-5R is used as the diameter monitoring device. It is capable of monitoring a .5 μm diameter change at a response rate up to 10 milliseconds. This particular model has been modified to output a 0 to 10 volt signal with a set value of 5 volts for nominal fiber diameter.

A galvanometric beam-scanning system (GSS) is used to generate a cylinder of energy from a $CO_2$ laser beam produced from a Model 41 Coherent $CO_2$ laser. The GSS has several advantages over the rotating lens system that is conventionally used including a 10-fold increase in operation frequency, (up to 500 Hz), accurate control over the neck geometry, and reduced silica vaporization rate. As a result, excellent diameter control is maintained.

An ultra-violet light is used for curing the plastic buffer that is applied as the fiber exits the HCS. This unit has dual air cooled curing heads each with variable power output control so that multiple plastic coatings can be applied to the hermetic coated fiber if desired.

The buffer coating aids in protecting the hermetic coated fiber from the microbending problems that have been noted by others. The hermetically coated and plastic buffered fiber is then spooled using conventional techniques.

A constant temperature water source is available for cooling the conical mirror, laser cavity, and vacuum pumps. It provides 16°C water at a rate of 35 gpm and pressure of 50 psi. The chiller unit of this rated capacity (178,000 BTU/hr) is necessary to stabilize the laser power delivered to the molten draw zone.

## Theoretical and Practical Considerations

The theoretical strength of silicate glasses depends on Young's Modulus (tensile modulus), the density of the silicon-oxygen bonds, and the strength of these bonds (Kao, 1980). For fused silica glass, the theoretical tensile strength approaches $2.6 \times 10^5$ psi. The actual short term strength of the glass fiber, however, is much less than its theoretical strength. Griffith attributed this reduction in strength to the presence of flaws or inhomogeneoties on the surface and/or in the substrate which act as stress concentrators. In addition, optical fiber exhibits delayed failure (static fatigue) and a wide variability in fracture strength. The long term reduction in strength is due to the stress induced, environmentally accelerated, growth of existing surface flaws to dimensions critical for spontaneous fracture. Variability in fracture strength occurs because of the size of the initial flaw causing the failure is statistically distributed.

The short-term strength of optical fiber is improved by eliminating internal defects and by reducing the size and density of surface flaws. In step and graded index and single mode fibers, the internal defects are obviously concentrated in either the core or the cladding of the fiber. Defects in the core are rare, but may include chemical

inhomogeneoties from contamination of the deposition gases. Clad defects are generally either seeds (bubbles), inclusions and/or air lines (linear air inclusions). By using high quality synthetic tubing, and high purity filtered deposition gases, these internal defects can be eliminated. Fiber strength then becomes a function only of surface conditions. Furthermore, the strength of graded, step and single mode fiber will be similar if identical deposition tubing (fiber cladding) and fabrication techniques are used. We, therefore, proposed to limit all fabrication drawing and optical and mechanical testing to silica and graded index fiber. This is done to limit the amount of testing that is necessary and to provide relevant mechanical and optical test data on the type of fiber which is presently most suited for long-term military applications. Other fiber types will be evaluated when indicated.

The two principal types of surface defects are scratches and deposited (imbedded) surface contamination. These stress concentrators may be largely eliminated through proper fabrication and handling techniques. Imbedded contamination has been greatly reduced by fabrication in clean room hoods. Proper pre-draw preform treatments have further reduced surface contamination. This involves etching in an HF acid solution, followed by flame polishing in a clean room hood, using glass tipped burners. Further environmental contamination and chemical corrosion are minimized by either drawing the preform immediately or storing in an inert, dust-free environment. The fiber draw environment is critical to the ultimate strength of the fiber.

These preform fabrication, pre-draw preparation and fiber draw improvements will be used in the data collection phase of this program.

The low loss, step or graded-index fibers will be coated with a urethane based polyacrylate. The resin is applied to the pristine surface of the fiber and cured in-line by ultra-violet radiation.

## E. Results and Conclusions

Evaluation of the system and technique for hermetically coating optical fibers is accomplished by measuring the performance and properties of the hermetically coated fiber compared to the uncoated fiber. This provides base line performance data as well as a control procedure to determine effects of variability in the hermetic coating. The hermetic coating is also evaluated independently to determine performance aspects and survivability of the basic coatings.

After installation and start up of the laser draw tower, initial runs were made on flame-polished silica preforms which indicated proof strength in excess of 100,000 psi. Initial fiber parameters considered at this point in time were relative flaw distribution, diameter variation, and concentricity in addition to static fatigue strength. The initial runs were spotted with many flaws. This is primarily due to handling difficulties using the available grade rod and contamination of the draw site area (since there was construction in progress at the time). Subsequent runs which will use flame-polished optical grade preforms are anticipated to show very low flaw distribution. Early runs have also indicated some difficulty with respect to diameter control and concentricity. These difficulties are due to a combination of factors, the most dominant being the nature of the laser heat zone at the low draw rates. Tests performed on fiber which have been drawn at rates in excess of 10 meters/minute indicated diameter control to be far superior - on the order of 1%. At lower draw rates, problems have been encountered using a feedback loop for diameter control. In this system, diameter information from the MilMaster is used to control the rod feed into the heat zone. At draw rates of several meters/minute, diameter variations on the order of 10% have been encountered. The control system is currently being modified to correct this problem by reducing the lag time between diameter sensing and the draw down zone. Adjustments to the galvanometric scanning mirrors to modify the length and concentricity of the heat zone are in progress. This will also assist in achieving greater diameter control due to the longer draw down region. Some minor problems were also encountered due to a surging problem in

the down feed system. This, however, only occurs during the start up procedure producing a lump in the fiber which can cause sticking when passing through the coating system ports. This problem, however, is readily circumvented by manual start up procedures. Additionally, problems have been encountered with power fluctuations in the $CO_2$ laser system. Most of these difficulties have been related to cooling problems and currently are being corrected by the addition of a high pressure water pump. As a result of these initial start up efforts, the strength and uniformity of the glass fiber has been greatly enhanced. Proof tests are now in excess of 300,000 psi and diameter variation has been reduced to $\pm$2%. Currently, problems are being addressed with respect to preform fabrication for the production of optical waveguides with reduced flaw distribution.

While the laser draw system was being evaluated, the hermetic coating system was also installed and made operational at GEOC. Initial problems encountered were detection and sealing of vacuum leaks, and increasing throughput capacity for some of the larger pumps. The throughput capacity was increased by enlarging the diameter of tubing for the primary mechanical pumps. Following these changes, the coating apparatus was able to achieve the desired vacuum levels for successful coating.

Once the vacuum system was fully operational, the HCS was aligned with respect to the draw axis. This consisted of aligning the upper and lower pumping stacks with respect to the fiber path through the HCS. After initial alignment, the coating apparatus was aligned with a low power helium-neon laser mounted below the HCS. The beam was required to illuminate the draw down zone of the rod, in order to verify alignment.

Following completion of the alignment phase, a 5 mm supersil rod was loaded for initial evaluation of the coating system. For these initial runs, carbon electrodes only were used so that the ion deposited carbon film would be the only coating on the fiber. Results from these tests indicated some problems existed due to oscillation of the fiber and system alignment which resulted in contact between the filter and the entrance and exit ports. It was found that with a slight variation of draw tension, the vibration of the fiber could be greatly reduced.

The initial runs also indicated a significant amount of flaws on the fiber. Some of these were caused by handling of the supersil rod and others by debris at the entrance port.

To assess the mechanical properties of the fiber, a static fatigue test system was constructed. The system is designed to evaluate 1 meter gauge lengths which can be strained at rates of 1.0, 0.1, and 0.01 meters/second. Preliminary tests indicated design modifications which were necessitated by the need to prevent fiber breakage at the anchoring pulleys. These problems are currently being corrected by using a new grooved pulley system with elastomer lined pulleys. The system is also now enclosed in a draft-free box to further eliminate testing variables. Once these initial problems have been corrected, evaluation of the hermetic coating system is anticipated to proceed quickly.

During the development of the hermetic coating process at ASL, initial tests and observations were made on glass fiber as well as slides which had been coated in the HCS. Since the only fiber available was bare fiber which had been exposed to atmosphere for several days, the characterization of fiber strength would be meaningless. However, other aspects of the coating could be determined; such as coating continuity, thickness, bond strength, and deposition rate. For the various materials considered, i.e., ion deposited carbon, indium, and tin, assessment was made as to film continuity. Coating runs performed on bare fiber with ion deposited carbon produced transparent coating with a slight brownish coloration. These coatings ranged in thickness from several hundred Angstroms to approximately 1,000 Angstroms. The coating was difficult to characterize due to its extreme thinness and transparency. Attempts at SEM were unsuccessful due to the high transparency of the carbon. In order to determine the presence, thickness, and continuity of the carbon film, it was necessary to remove the film from the fiber by dissolving the glass fiber in hydrofluoric acid. Once this was done, the carbon film could be removed as a free-floating thin film and transferred onto the glass slide for examination. A sample of this film is shown in Figure 5. The film showed itself to be continuous with sufficient strength to maintain its integrity in thicknesses of several hundred Angstroms. Films of

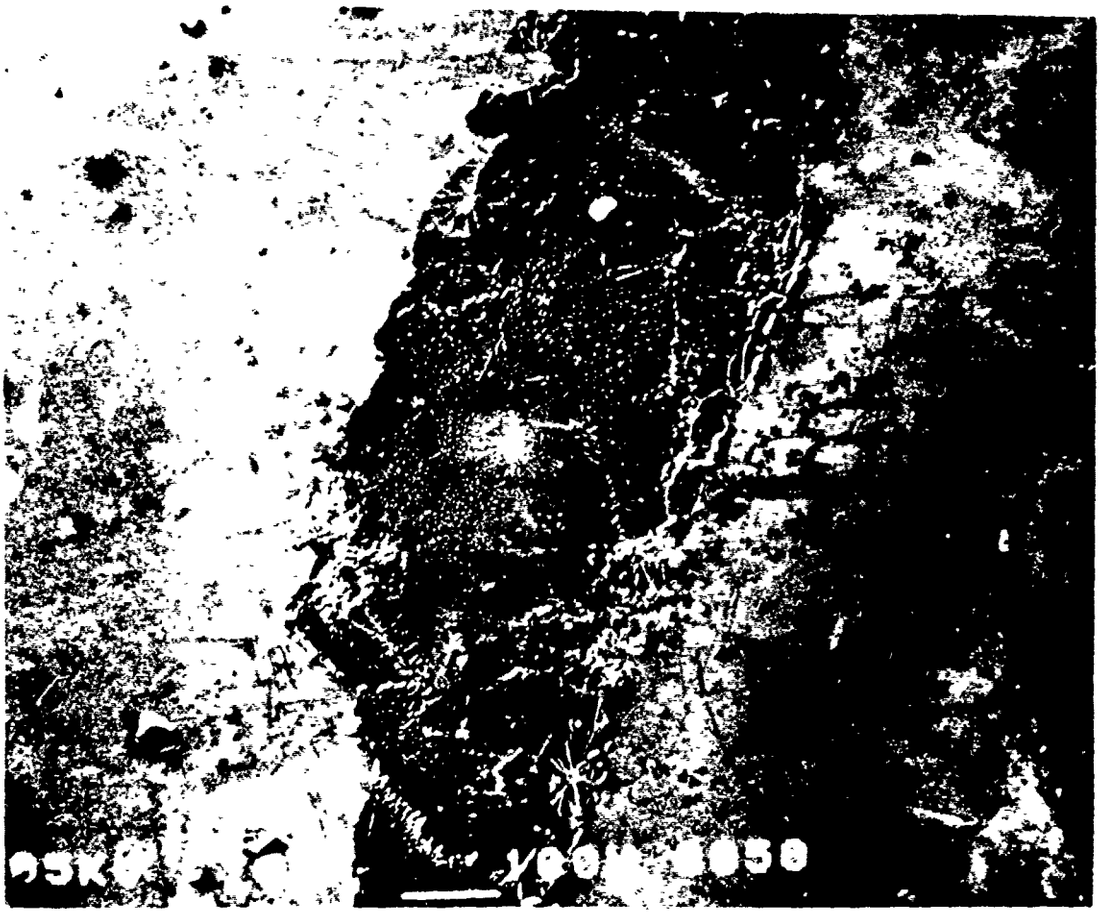

Fig. 5 Ion Plasma Deposited Diamond-Like Carbon Film
With Fiber Dissolved Away - Film Unfolded Onto Slide

indium on carbon were also evaluated. Fortunately, these could be observed much more readily than the carbon film. The nature of the deposition appeared to be the same for indium as well as tin. The majority of subsequent work was based on indium due to its low ductility and zero memory. Initial results using the indium indicated spotty globular coatings were being produced following SEM evaluation of the films. Consequently, steps were taken to increase the ratio of metallic ion to atom density thereby gaining greater control over surface coalescence once the indium encounters the fiber surface. The results of this phase indicated a more continuous coating which appeared metallic and shiny rather than dull and granular (see Figure 6). Adhesion determination was difficult using the fiber since bond strength measurement could not be made. Consequently, tests for the ion deposited carbon were performed on glass slides indicating bond strengths in excess of 2,000 psi which appears acceptable for the intended application.

Fig. 6 Smooth Surface of Indium Film as Deposited On Fiber - Viewed Through Polymeric Buffer Coat

## IV. WORK REMAINING

At the time of this Report, in-line fiber drawing and coating has been in progress for several weeks. We are beginning the dynamic and static testing portion of this program to assess fiber characteristics including Weibull statistics and regression parameters. The program will continue through a series of fiber runs and testing series, followed by modification to the system and subsequent runs.

Initial problems identified with the first coating runs include difficulties with the polymer coating system, movement of the fiber causing occasional contact at the entrance and exit ports of the hermetic coating apparatus, and dust and debris adhering to the fiber upon entering the system.

These problems will be addressed immediately since valid statistics are difficult to generate otherwise. The fiber path will be enclosed with a gas shroud, thereby preventing extraneous dust and debris from the room from adhering to the fiber prior to coating. Additionally, the entrance apertures to the coating system will be modified to reduce dust adhesion as well as reduce fiber contact. A laminar flow system will be provided to eliminate surface dust. The room housing the draw tower is also in the process of being enclosed. This work has created considerable dust producing significant variations in fiber strength.

Once the coating parameters and test technique have been stabilized, longer drawing runs will be made both for the indium and carbon to assess key deposition parameters which would include deposition pressure, draw rate, electrode geometry, and electrode potentials, as well as material thickness and materials composition. These parameters will be changed based on the experimental results determined, based primarily on fiber strength. Additionally, study of the nature of the fracture zone from dynamic strength tests, as well as analysis of the coating by various techniques will be carried out.

## V. CONCLUSIONS

Strength results during the start up period were lower than expected due to contamination of the draw site. This resulted in reduced strengths for laser drawn fiber with no coating applied. Strengths were further reduced as debris was pulled into the coating system with the fiber. These results indicate the importance of cleanliness if high quality fiber is to be drawn. The rennovation of the draw area is nearly complete so we may expect results typical of laser drawn fiber shortly.

More importantly, however, has been the verification of the in-line ion deposition system performance. Coatings of carbon 600 Angstroms thick and indium 2 microns thick have been deposited at draw rates to 8 meters/minute. Since coating rate is directly proportional to coating electrode length, production draw rates may readily be achieved. The current combined electrode length is now only 15 cm for deposition of both materials. SEM observation of the carbon film indicates it to be continuous and pinhole-free as is also to be expected of the indium film.

Results to date indicate that the energetic ion deposition process is a viable method for producing thin films on glass fibers. Extension to optical waveguides should present no problems since the outer clad is the only zone of the fiber affected by the hermetic coating process. Major efforts, for the remainder of the program will be the testing of the fiber performance, both mechanical as well as optical, as well as characterization of flaw distribution, fiber diameter, concentricity and attenuation. The coating will also be characterized for an improved resistance to static fatigue which is the major effort of this program. Additional tests will be performed to assess the nature of microbend losses as well as evaluation of other degradation mechanisms.

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# APPENDIX A

## SUMMARY OF OBSERVED PROPERTIES OF ION-DEPOSITED DIAMOND-LIKE CARBON FILMS

## SUMMARY OF OBSERVED PROPERTIES OF ION-DEPOSITED DIAMOND-LIKE CARBON FILMS

The following is a summary of the various properties of diamond-like carbon as deposited using the Ion Beam Plasma Deposition Process first described by the staff at G-W Applied Science Laboratories.[1] These properties were determined by Applied Science Laboratories and by other workers in this field.[2] Some of these results are based on limited observations.

(1) Optically Transparent - Water clear or pale yellow. Low IR absorption in thin film form (0.3 to 10μ)

(2) Insulating - $10^{11}$ - $10^{12}$ ohm-cm AC and DC resistivities. May be less insulating at faster deposition rates.

(3) Good adhesion to substrate - Pull test measurements on metal substrates showed up to 5,000 psi adhesion.

(4) Can be deposited on various substrates - (at about 10°C above room temperature) - has been deposited on glass, silicon, mylar, paper, NaCl, KCl, germanium, plexiglass, stainless steel, and a wide variety of other substrates.

(5) Chemically inert to all known solvents - these include HF, $HNO_3$, $H_2SO_4$, HCOOH, bases, acetone, trichloroethane, chloroform, and ethyl acetate.

(6) Film is unusually smooth - scanning electron microscope shows film is smoother than substrate. This is probably due in part to the ion energy, and the electrical charge on the surface, modifying the surface tension.

(7) Apparent reduction in surface optical scattering - reports indicate that the film increases both transmission and reflection at the substrate surface. This suggests that the smoother surface of the film reduces surface scattering. The ion bombardment cleaning of the substrate during deposition can also remove absorbing surface impurities. The inert carbon film should adsorb less impurities and water monolayers.

(8) Film is free of pinholes - due to electrical charge on surface, and high ion energy

(9) Film is diamond-like - amorphous and/or polycrystalline, with crystallites in the 50-100Å range according to X-ray diffraction line broadening. Definite lines assignable to diamond have been reported. Films show a cubic lattice with lattice constant close to those for cubic diamond.

(10) Low impurity content - ion backscattering and Auger studies show a few percent of carrier gas (argon). Hydrocarbons in films were reported when hydrocarbons were added to carrier gas. Oxygen was reported on surface (true of all surfaces exposed to air) but oxygen was not found in bulk.

(11) Hard - films are very hard as shown by scratch tests

(12) Density - 2.36 $gr/cm^3$

(13) Low porosity

(14) Moisture and gas barrier

(15) High index of refraction - about 2 in visible range, from ellipsometric measurements

(16) High dielectric constant - about 16, from capacitance measurements

(17) Erosion resistance - reported to be good, from rain erosion tests

(18) Low surface potential - about 0.1 volt negative, as determined by Kelvin vibrating electrode method

(19) High dielectric breakdown voltage - $10^6$ volts/cm from measurements

(20) Film thickness is normally made to be about 800Å, but has been made up to 10μ. For thicker films, the films may separate from substrate, probably due to difference of thermal expansion coefficient.

(21) Free standing films obtained by dissolving substrate are flexible

(22) There is no curl in free standing film, showing an absence of built-in stress.

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(1) Aisenberg, Sol and Chabot, Ronald, Ion Beam Deposition of Thin Films of Diamond-like Carbon, Journal of Applied Physics, Vol. 42, No. 7, June 1971.

(2) Spencer, E.G., Schmidt, P.H., Joy, D.C. and Sansalone, F.J., Ion-Beam-Deposited Polycrystalline Diamondlike Films, Applied Physics Letters, Vol. 29, No. 2, July 1976.



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## APPENDIX B

## SOLUTIONS TO POTENTIAL PROBLEMS

For the benefit of other workers in the field, we will list a number of potential problems and discuss reasons why the ion-plasma coating approach is expected to provide solutions to these potential difficulties.

1. Corrosion of Metal Coatings

Pure metals will be used in order to avoid the potential corrosion of dissimilar metals and alloys. High purity metals of 99.9% or better purity will be used since they are not prohibitively expensive. In particular, we will use metals that will have a reported high immunity to corrosion, such as indium or as tin. These pure metals also have the desired properties of being ductile so that one does not expect work hardening as a result of flexing. In addition, the metallic coatings on the glass fibers will be subsequently coated with the usual protective coating of plastic which will act as a barrier to gross migration of moisture. Even though water vapor can permeate through the plastic and react with the metallic surface, the reaction will be very low because of the absence of migration of ionic components, such as sodium or chlorine, through the plastic. Also, the rate of corrosion of the metal is determined by the rate at which corrosion by-products can be transported away. Since any corrosion, even if it did exist, would occur under a protective plastic coating, the corrosion by-products would be trapped and, therefore, any corrosion of the metallic coating would be self-limiting. We, therefore, do not anticipate any corrosion because of:

a. The corrosion resistance of the metallic coatings selected;
b. The barrier properties of the protective plastic coating limiting the transport of water and salts to the metallic surface;
c. The barrier properties of the plastic limiting the transport of the corrosion ions away from the metallic surface;
d. The purity of the film preventing local galvanic reactions.

2. Hydrogen Embrittlement

There is a possibility that metallic films deposited in a hydrogen atmosphere can contain sufficient hydrogen so that the metals become brittle. This could cause difficulties under the flexing conditions anticipated. We propose to eliminate this problem by using ductile metals, such as tin or indium, which are not subject to hydrogen embrittlement, while at the same time perhaps avoiding the use of hydrogen during the deposition process, except through decomposition of trace amounts of water vapor. The release of metal vapor into the electrical discharge accompanying deposition of the metal will act as a very effective getter for trace amounts of contaminating gases such as hydrogen, since larger amounts of this metal will also be deposited on the chamber walls enclosing the fiber being coated. We do not anticipate any difficulties due to hydrogen embrittlement as a result of the approach we are taking.

3. Effect of Ion Milling on Fiber Strength

One of the advantages of the process is that it uses a sequence of ion bombardment and ion sputtering for removal of surface contamination of the fiber as the fibers are drawn through the deposition chamber. It is not anticipated that the ion milling will reduce the basic fiber strength since we do not anticipate removing more than a few monolayers of the surface. If the fiber itself is defective because of the inclusions within the fiber, then this process we are applying is not expected to correct major defects. The primary benefit of the ion milling is to remove small surface defects which could grow into larger defects as a result of stressing. The ion milling also removes contamination layers which would interfer with the adhesion of the subsequent metallic coating to the glass fiber.

4. Surface Inclusions in Fiber

If there are surface inclusions in the fiber as a result of surface defects in the basic glass rods during drawing, then this ion

milling and metal film deposition process will not remove these inclusions but will protect these inclusions from encountering water vapor which might make the difference between failure or survival of the fiber at these minor inclusions. Major inclusions, of course, would fail with or without the presence of water vapor.

5. Flexing and its Effect on Film Creep

The system is designed to put down a ductile adherent film of metal to act as a protective barrier against moisture. The cyclic elongation and flexing of the basic fiber will also result in the cyclic flexing of the metal coating. The relative ductility of the pure metallic coating compared to that of the glass substrate means that the metal would have a putty-like consistency and can flow and reflow as a result of flexing of the substrate. These metals are chosen to be good oxide formers and will bond quite well to the glass fibers, particularly in view of the ion bombardment cleaning of the substrate during the deposition of the priming metallic coat. The metals are chosen to be pure and ductile so no work hardening is anticipated, and the metals will remain ductile. The ductility of the metal and the flexing is an advantage in the sense that it will tend to reduce any pinholes that may appear in the metal. Although we do not anticipate pinholes in the metal, if there were pinholes, and if the fiber and metallic coating were flexed, the metallic material would tend to flow in and fill the void in the metallic film, since there is no material there to provide restoring forces preventing such flow. Therefore, one would expect that a ductile metallic coating adherent on a glass fiber substrate with a pinhole in the metal film would have the size of the pinhole reduced as a result of a number of cycles of flexing.

6. Work Hardening

Since the fiber and metallic coatings are expected to be exposed to flexing cycles, it is desirable to use a metal film which is resistant to work hardening. Pure metals such as indium and tin are much more

resistant to work hardening than alloys. The ductile nature of the metal prevents transmission of metal defect forces into the glass.

7. Water on Surface of Fiber

It is possible that the fiber entering the deposition coating system can have monolayers of water vapor on the surface. One wishes to be sure that the water vapor is not trapped under the metallic fiber. The process being used is designed to avoid that problem since the fibers pass into a low pressure chamber where the surface is bombarded with high energy plasma-ions. This will tend to scrub the surface water molecules off and there will be no source of water vapor for replacement. At the same time, when the fiber enters the metallic deposition chamber, the fiber surface is again bombarded by energetic ions simultaneously with the deposition of the metallic priming film. The temperature of the fiber will be moderately high and this will tend to also reduce the coverage of any residual water vapor. The continuous vaporization of the metal onto the walls of the metallization chamber will provide a large area getter which will react with any residual adsorbant gases and water vapor in the system and trap them rapidly.

8. Surface Dust

It is possible that the fiber in passing from the fiber drawing chamber to the deposition chamber may have dust or lint particles on the surface. We can use a region of moderately pressurized air circulating in a vortex configuration to scrub any loose dust particles from the surface of the fiber before it passes into the differential pumping port. Any dust that is fused into the surface of the glass fiber, of course, will not be readily removed. We can use a pressurized gas shroud connecting the entrance of the vortex scrubbing chamber to the exit of the fiber drawing chamber. The use of a pressure in excess of atmospheric pressure will provide a counterflow which will keep outside ambient air from entering along with any potential dust particles.

## 9. Adhesion of the Metallic Film to the Glass Fiber

In normal evaporation of the metallic films onto glass, there is sometimes a problem of adhesion. This is particularly true of the noble metals, such as gold or silver, and is also partly due to layers of gas on the surface during the evaporation period. In this plasma-ion deposition technique the surfaces are cleaned of contaminating layers during the actual deposition and this factor will improve the adhesion. In addition, the surface is bombarded with charged particles (ions and electrons) and energetic atoms during the deposition. This form of deposition with the simultaneous plasma exposure has been reported by others to give unusually good adhesion. In addition, we are using oxide forming materials such as indium or tin, which in themselves regularly give good adhesion, even to surfaces which are not exposed to plasma conditions. The use of oxide formers is expected to be of considerable help in providing adhesive metallic films.

## 10. Lateral Migration of Moisture Vapor from Pinholes Along Metal Glass Interface to Surface Defects

In reviewing this process, one should consider even the less likely case of pinholes in the film plus the presence of the stress concentrating defects of the substrate glass fiber. It is anticipated that statistically the location of a pinhole in the metal film will not correspond to the location of a defect in the substrate glass. In order for moisture to take advantage of this pinhole and arrive at the defect in the glass, a very unlikely process would be required. One would require that water molecules would travel through the pinhole to arrive at the glass surface and then laterally transport themselves along the interface between the metal film and the glass surface in all directions, with one of these directions corresponding to that of a remotely located glass surface defect. The expected transport of water molecules at the interface of metal glass is very unlikely, particularly in view of the fact that one has an oxide forming metal on the glass, which adheres very well to the glass. Only in the event

that there were severe delamination problems between the metal film and the glass would one expect to see this lateral transport of water vapor. These defects could serve as a channel for transport of water molecules laterally. The method we are proposing should be free of this possible defect because of the plasma-ion cleaning of the surface and the ion-plasma deposition process.

## 11. Entrance and Exit Ports

There is the necessity for insuring that the glass fiber in entering the differential pumping aperture is clear of the walls and does not touch them on the way into the vacuum treatment and vacuum metallization chambers. The problem of contact with the wall on the way out is less critical since the surface, at that point, is protected with a metallic film. The fiber will be about 5 mils in diameter and will proceed down the center of the channel. It is important to realize that if the alignment is not completely correct the fiber can touch the walls of the channel. For this reason we are making the channel walls of material that will not tend to damage fiber in case of accidental contact. This is to protect fiber during initial alignment. During actual drawing there will be little or no contact. For this purpose we will use teflon or hard smooth metal inserts as bushings in the differential pumping ports to provide the 60 mil diameter channel and also to facilitate the fabrication of the multi-stage differential pumping apertures.

## 12. Alignment

The positioning alignment can be taken care of by careful operator observation during set up, plus the use of an optical sensor which can detect the position of the fiber with respect to a light source and detectors mounted on the entrance and exit ports.

In addition, a laser alignment procedure can be used in interfacing the fiber drawing system and the hermetic coating system.

# # #

# APPENDIX C

## SUMMARY OF PROPERTIES FOR INDIUM

## SUMMARY OF PROPERTIES FOR INDIUM*

| | |
|---|---|
| Atomic weight | 114.82 gm |
| Crystal structure | Face-centered tetragonal |
| Density | 7.31 gm/cc at 20°C (68°F) |
| Electrical resistivity (ohm-cm) | |
| Latent heat of fusion (cal/g) | 6.807 |
| Latent heat of vaporization (cal/g) | 468 |
| Linear coefficient of thermal expansion per 1° C/cm | $24.8 \times 10^{-6}$ |
| Magnetism | Diamagnetic |
| Mechanical properties | |
| Tensile strength, psi | 380<br>515 |
| Compressive strength, psi (True compressive stress at 10% true strain) | 310 |
| Elongation (% in 1") | 22<br>41 |
| Brinell hardness | 0.9 to 1.0 |
| Reduction in area (%) | 85 |
| Melting point | 156.61° C |
| Boiling point | 2080°C<br>(3632°F) |
| Solidification shrinkage | 2.5% |
| Specific heat (cal/g/°C) (Solid) 20°C | 0.065 |
| Specific volume (cc/g) 20°C (68°F) | 0.136 |
| Surface tension (dynes/cm°C) 170°–250°C | 340 |
| Surface tension at MP dynes/cm | 513; 599 |
| Thermal conductivity (cal/sq. cm/cm/°C/sec) 20°C | 0.17 |
| Valence | 3 also 2 and 1 |
| Vapor pressure (mm Hg) | |
| 1249°C (2280°F) | 1 |
| 1466°C (2671°F) | 10 |
| 1756°C (3193°F) | 100 |
| 1863°C (3385°F) | 200 |
| 1982°C (3600°F) | 400 |

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